Development of phthalate-free quartz discs that prevent scattering of powdered samples out from Eco-Cup

Part 1: Effect of quartz disc and comparison with quartz wool

[Background] In pyrolysis GC measurements, in which a sample in a sample cup (Eco-Cup) is introduced into the furnace by the free-fall method, quartz wool is placed on the top of the sample to prevent scattering of a powdered sample in the furnace. However, placing wool into Eco-Cup is cumbersome, and the form and amount of the wool may vary between analysts, which can affect the reproducibility of analysis results. Therefore, an easy-to-handle phthalate-free quartz disc (quartz disc; Fig. 1) for preventing sample scattering was developed. In this study, the effect of the presence or absence of the quartz disc in the measurements of polystyrene (PS) containing phthalates was evaluated.

[Experimental] A GC/MS system equipped with a Multi-Shot Pyrolyzer directly interfaced to the GC injector was used for measurements. Quartz discs were prepared by punching out a quartz filter paper into a circular shapes with a diameter of 3 mm, followed by deactivation treatment. As a sample, a dichloromethane and xylene (9:1) solution of PS (50 µg/µL) containing 1000 ppm each of seven phthalates specified in IEC 62321-8 was prepared, and 5 µL of the solution was put in an Eco-Cup. The solvent was allowed to dry at room temperature. Then, one quartz disc was placed in the Eco-Cup, and the sample was measured by thermal desorption-GC/MS.

Fig. 1 Photo of quartz disc.

[Results] Fig. 2 shows the chromatograms obtained from measurements with quartz wool (1 mg) and with quartz disc. The results indicate that the chromatograms are equivalent. Table 1 summarizes the recoveries of phthalates with quartz disc (normalized against those using quartz wool) and the reproducibilities obtained from measurements (n=3). Nearly 100% recoveries and excellent reproducibilities were obtained for all phthalates. The results demonstrate that the quartz discs have no impact on phthalate analysis.

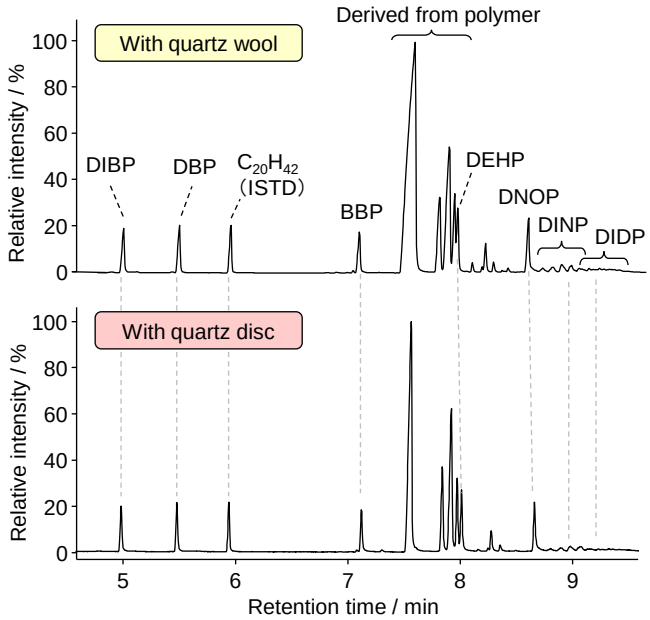

Fig. 2 Chromatograms obtained with quartz wool and quartz disc.

Furnace temp.: 200 - 20 ºC/min - 340 ºC (1 min hold), Separation column: UA-PBDE (*L*=15 m, i.d.=0.25 mm, df=0.05 µm),
Column flow rate: 1 mL/min, Split ratio: 1/20, GC oven temp.: 80 - 20 ºC/min - 300 ºC (5 min hold),
MS scan rate: *m/z* 29 - 450, Sample amount: 0.25 mg (PS).

Table 1 Recovery and reproducibility (n=3) of seven phthalates with quartz disc

	Recovery [%]	RSD [%]
DIBP	107	0.7
DBP	105	1.2
BBP	100	0.9
DEHP	104	0.6
DNOP	100	2.9
DINP	106	1.9
DIDP	107	1.1

Keywords : Sample preparation method, Phthalates. RoHS2.0, RoHS directive, thermal desorption

Products used : Phthalate free quartz disc, Eco-Cup LF, Multi-Shot Pyrolyzer, Auto-Shot Sampler, UA-PBDE, Vent-free GC/MS adapter

Applications : General polymer analysis, Sample preparation method

Related technical notes : PYA1-153E (Part 2)

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Frontier Laboratories Ltd.
Phone: (81)24-935-5100 Fax: (81)24-935-5102
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